CHAPTER V

RESULTS AND DISCUSSION

In this section, results from process screening using the visual inspection results and the sidewall cleanliness results based on the SEM audit are discussed. The process utilization was also considered and the selected cleaning condition was compared with the current base recipe in terms of cleanliness, electrical performance and defect failure. Referred to Figure 2.9 in Chapter II, this research focused on the NaOH cleaning process by taking into account of NaOH concentration and scrubbing time only.

5.1. Screening process recipe

For screening the process recipe, fist step was to study the pitting defect and roughly checking for the etch sidewall cleanliness. Pitting defect is the side effect of this process which can be detected by visual inspection. Pitting defect rate has been shown to increase with increasing NaOH concentration and scrubbing time. The result of visual inspection showed the limit of scrubbing time that can be used.

Table 5.1 Pitting defect due to scrubbing versus sodium hydroxide concentration.

NaOH concentration (%w/v)	Increasing after 4 min	Increasing after 8 min	Increasing after 12 min	Increasing after 16 min	Increasing after 20 min
0.02%	0.00%	0.00%	0.00%	0.00%	2.29%
0.05%	0.00%	0.00%	0.00%	2.28%	3.58%
0.10%	0.00%	0.00%	0.91%	7.47%	8.54%
0.30%	0.00%	1.07%	7.16%	8.38%	13.57%
0.50%	0.00%	7.01%	9.60%	14.33%	21.04%
1.00%	4.88%	10.52%	19.51%	30.49%	38.57%

As shown in Table 5.1, with low concentration, the scrubbing can be applied to the part at a longer time than at higher concentration. At 1% NaOH concentration, the pitting has occurred since the first 4 min of scrubbing. While at 0.5% and 0.3%, pitting was both seen to start after 8 min of scrubbing. For 0.1%, 0.05%, and 0.02%, pitting did not appear until scrubbing at 12 min, 16 min, and 20 min respectively.

In order to find conditions that provide zero percent of pitting defect, experimental design was conducted to run with parts and check for cleanliness at etching sidewall. SEM was used to observe the etch sidewall cleanliness and check for process recipe screening. Table 5.2 shows the number of post-etched redeposit found on etched sidewall in each cleaning condition.

Table 5.2 Sidewall cleanliness inspection results subject to recipe screening.

Process Flow	Sample size (Qty.of SEM)	Found Redeposition (post-etched residue)
0.02%NaOH scrub 16 min	80	31
0.05%NaOH scrub 12 min	80	3
0.10%NaOH scrub 8 min	80	4
0.30%NaOH scrub 4 min	80	5
0.50%NaOH scrub 4 min	80	4
0.02%NaOH scrub 4 min (Current base recipe)	80	71

Based on the observation shown in Table 5.2, the condition of cleaning with 0.05% NaOH and scrubbing time of 12 min would result in the best redeposit removal. At 0.05% NaOH with 12 min scrubbing time, the lowest residue was found after compared with the other cleaning condition. Also under such condition, the manufacturability is better for the following reasons:

1) Minimal increase of operation cost;
2) Ease of handling and better safety because this is the lowest concentration of NaOH; and
3) Required no additional machine modification such as piping and waste treatments.

There were 2 outputs to measure: 1) part cleanliness which contains part cleanliness by SEM, AFM and the anion residue check by IC. 2) electrical yield and failure which is related to part cleanliness.

5.2. Parts cleanliness comparison

5.2.1 Comparison of Etch Sidewall Cleanliness Comparison Using SEM

By using SEM for sidewall cleanliness inspection, the result as shown in Figure 5.1 showed that the post-etched residue from new 0.05% NaOH scrubbing with 12 min had 5 times lesser redeposit rate than the current 0.02%NaOH scrubbing with 4 min.

Process Flow	Sample size (Qty.of SEM)	Found Redeposition (post-etched residue)	Defect Proportion
0.05%NaOH scrubbing 12 min	243	49	0.2016
Normal (0.02%NaOH scrubbing 4 min)	264	231	0.8750

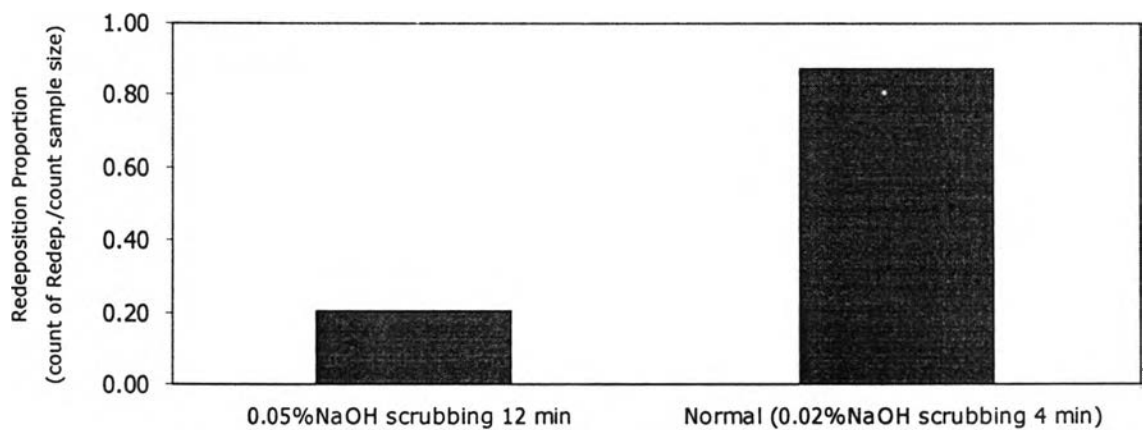


Figure 5.1. A comparison of the post-etched residue (redeposit) inspection level.

From the count of detected residue, the defect proportion was calculated. The proportion of post-etched residue of 0.05%NaOH process and 0.02%NaOH process was 0.2016 and 0.8750, respectively. Based on the defect proportion, we can apply the standard statistical analysis of 2-propotions testing (see the testing procedure in appendix A) to compare the effectiveness of post-etched redeposit.

Test and CI for Two Proportions – Post etched residue (Redeposition)

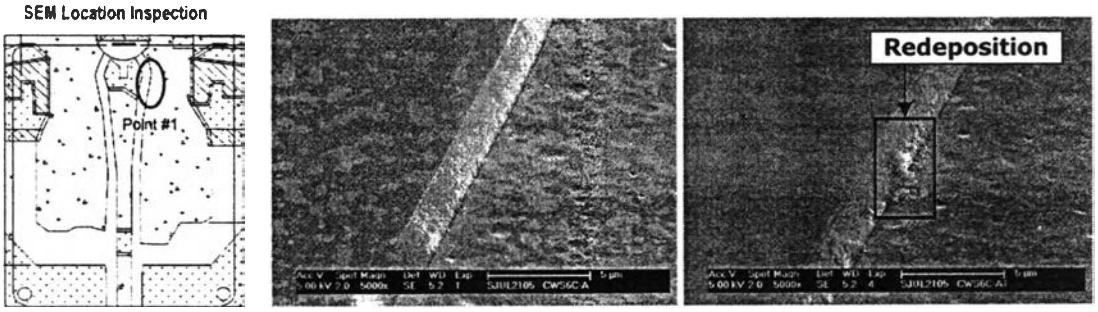
```
Sample                                  X    N  Sample p
0.05%NaOH scrubbing 12 min             49  243  0.201646
Normal(0.02%NaOH scrubbing 4 min)     231  264  0.875000

Difference = p (1) - p (2)
Estimate for difference:  -0.673354
95% CI for difference:  (-0.737669, -0.609039)
Test for difference = 0 (vs not = 0):  Z = -20.52  P-Value = 0.000
```

removal as shown in Figure 5.2.

Figure 5.2. A print-out of statistical analysis on redeposit removal from Minitab.

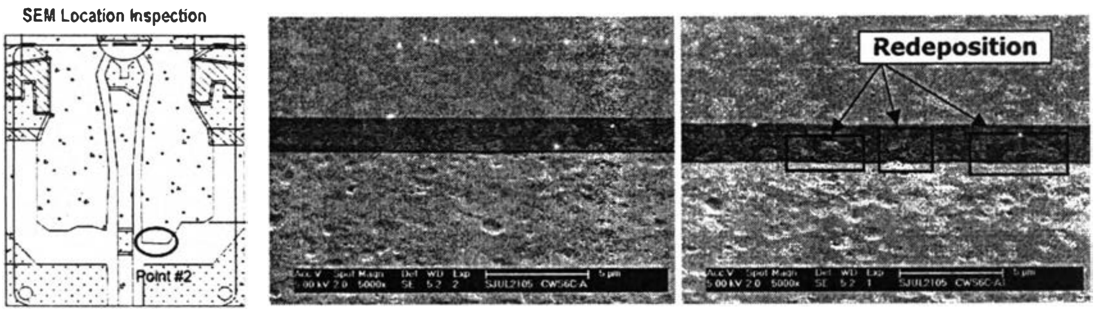
The P-value from this analysis showed less than 0.05 at 95%confidence level. This suggested that in terms of potential defect percentage of both cleaning conditions are significantly different to each other considering the high confidence level applied with approximately 0.7 proportion gap. This data revealed that the post-etched removal of 0.05% NaOH scrubbing 12 min is significantly better than that the current condition with 0.02% NaOH scrubbing 4 minutes.

The SEM images in Figure 5.3 and 5.4 also showed parts with 0.05% NaOH 12 minutes scrubbing were cleaner than parts with 0.02%NaOH 4 minutes scrubbing.



(a) 0.05%NaOH with 12 min scrubbing (b) 0.02%NaOH with 4 min scrubbing

Figure 5.3. SEM images of point 1 sidewall that has been cleaned (a) with 0.05% NaOH 12 minutes scrubbing; and (b) with 0.02%NaOH 4 minutes scrubbing.



(a) 0.05%NaOH with 12 min scrubbing (b) 0.02%NaOH with 4 min scrubbing

Figure 5.4. SEM images of point 2 sidewall that has been cleaned (a) with 0.05% NaOH 12 minutes scrubbing; and (b) with 0.02%NaOH 4 minutes scrubbing

5.2.2 Comparison of Etch Sidewall Cleanliness Comparison Using AFM

Result of the AFM scan along the sidewall is shown in Figure 5.5. There was 3 times lower observation rate for spike defect at etch sidewall from new 0.05% NaOH scrubbing with 12 min scrubbing than current 0.02%NaOH scrubbing with 4 min.

Process Flow	Sample size (Qty.of AFM)	Found Spike (protruded residue)	Defect Proportion
0.05%NaOH scrubbing 12 min	240	13	0.0542
Normal (0.02%NaOH scrubbing 4 min)	255	33	0.1294

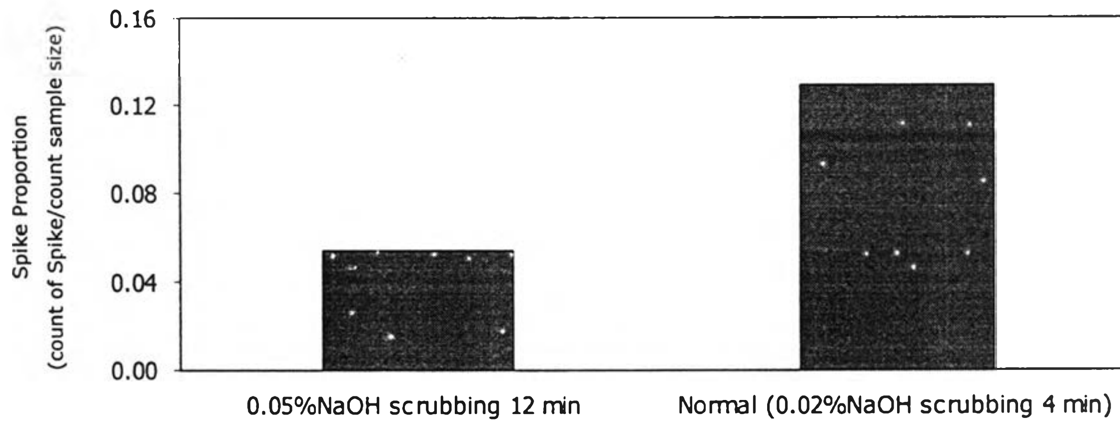


Figure 5.5. Comparison of the protruded residue –"spike" – inspection result

From the count of detected residue defect proportion was calculated for further analysis. The proportion of post-etched residue of 0.05% NaOH process and 0.02%NaOH process was 0.0542 and 0.1294, respectively. Based on the defective proportion, the standard statistical analysis of 2-propotions testing was made to compare the effectiveness of protruded residue (spike) removal.

Test and CI for Two Proportions - Protruded residue (Spike)

```
Sample                                     X    N  Sample p
0.05%NaOH scrubbing 12 min                13  240  0.054167
Normal(0.02%NaOH scrubbing 4 min)         33  255  0.129412


Difference = p (1) - p (2)
Estimate for difference:  -0.0752451
95% CI for difference:  (-0.125418, -0.0250727)
Test for difference = 0 (vs not = 0):  Z = -2.94  P-Value = 0.003
```

Figure 5.6. A print-out of statistical analysis on the spike removal from Minitab.

From statistical analysis in Figure 5.6, the P-value of this analysis was less than 0.05 at 95%confidence level. This suggested in terms of potential percent defect of both cleaning conditions were significantly different to each other given the high confidence level with approximately 0.07 proportion gap. This data revealed that the spike removal of 0.05 %NaOH scrubbing 12 min was significantly better than current 0.02%NaOH scrubbing 4 min.

The AFM images shown in Figures 5.7 to 5.9 revealed that the part after cleaning with 0.05% NaOH for 12 min was more effective in eliminating the spike defect than the 0.02% NaOH and scrubbing time of 4 minutes.

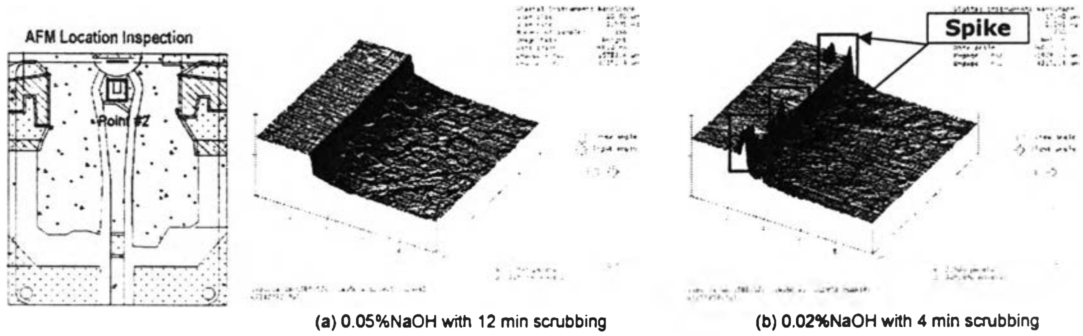


Figure 5.7. AFM images of point 2 sidewall that has been cleaned (a) with 0.05% NaOH 12 minutes scrubbing; and (b) with 0.02%NaOH 4 minutes scrubbing.

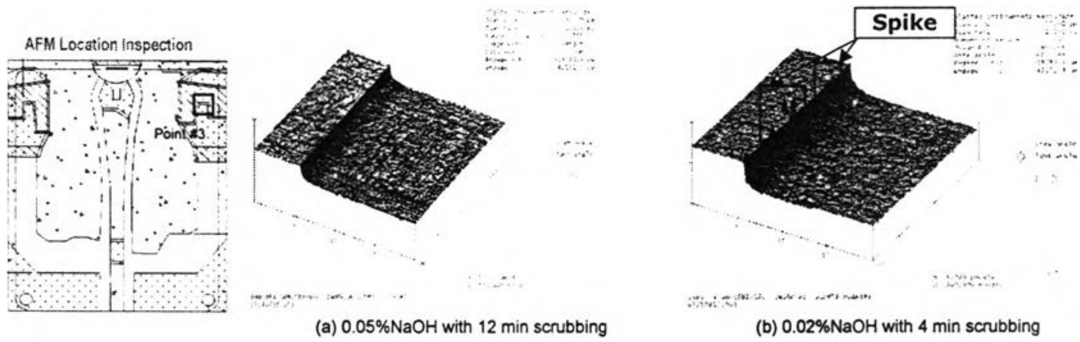


Figure 5.8. AFM images of point 3 sidewall that has been cleaned (a) with 0.05% NaOH 12 minutes scrubbing; and (b) with 0.02%NaOH 4 minutes scrubbing.

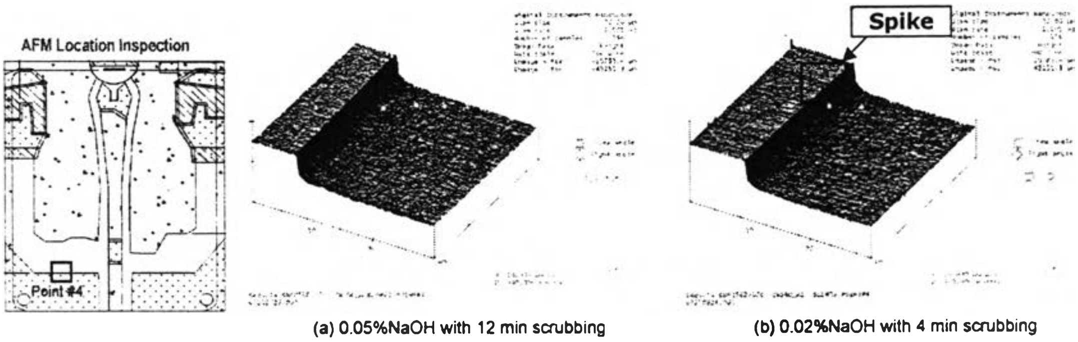


Figure 5.9. AFM images of point 4 sidewall that has been cleaned (a) with 0.05% NaOH 12 minutes scrubbing; and (b) with 0.02%NaOH 4 minutes scrubbing.

5.2.3 Comparison of Fluoride Ion Residues Using Ion Chromatography

The IC has been employed to chemically identify the post-etched redeposit. The result showed presence of the fluoride ion that came from CF_4 gas during the etching process. In Table 5.3, the fluoride ion (microgram/cm^2) left on the part from each trial of both cleaning conditions was summarized.

Table 5.3 Quantity of fluoride ion existing on cleaned parts after cleaning.

Group / Trial#	Fluoride ion existing on the part with 0.05%NaOH scrubbing 12 min (microgram/cm^2)	Fluoride ion existing on the part with 0.02%NaOH scrubbing 4 min (microgram/cm^2)
1	2.60	2.11
2	2.24	3.74
3	1.95	4.25
4	2.08	3.66
5	1.87	4.07
6	2.19	3.83
7	2.45	2.98
8	1.98	3.56
9	2.01	4.01
10	2.45	2.70
11	2.11	3.49
12	2.04	3.51
13	1.93	4.12
14	2.36	3.48
15	1.88	3.12
Average	**2.14**	**3.51**

Figure 5.10 is a print-out of the fluoride ion residue on the part after cleaning plot from Minitab. The plot showed average of fluoride ion left on the part after clean with 0.05% NaOH scrubbing for 12 minutes was lower than part after clean with 0.02% NaOH scrubbing for 4 minutes.

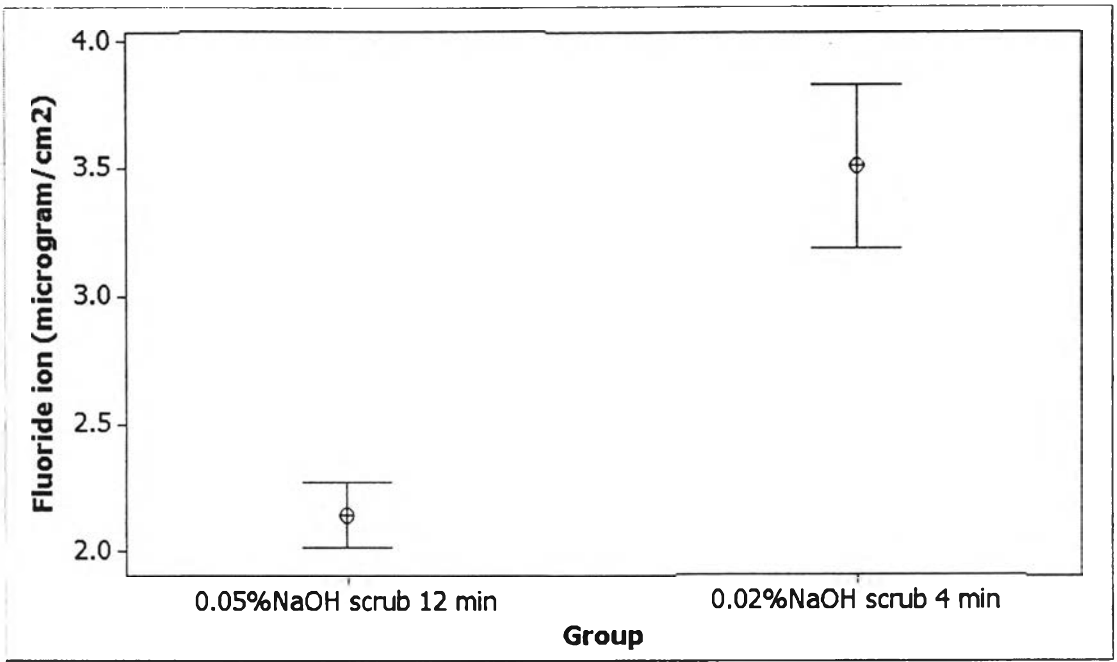


Figure 5.10. Plot of interval plot of fluoride ions on part from Minitab.

Two-Sample T-Test and CI: Fluoride ion (microgram/cm^2), Group

Two-sample T for Fluoride ion (microgram/cm^2)

Group	N	Mean	StDev	SE Mean
0.02%NaOH scrub	15	3.509	0.580	0.15
0.05%NaOH scrub	15	2.143	0.230	0.059

Difference = mu (0.02%NaOH scrub 4 min) - mu (0.05%NaOH scrub 12 min)
Estimate for difference: 1.36600
95% CI for difference: (1.02735, 1.70465)
T-Test of difference = 0 (vs not =): T-Value = 8.47 P-Value = 0.000 DF = 18

Figure 5.11. Print-out of statistical analysis on Fluoride ion residue from Minitab.

From the statistical analysis as shown in Figure 5.11, the P-value of this analysis was less than 0.05 at 95%confidence level indicating that in terms of potential fluoride ion residue of both groups were significantly different to each other considering in high confidence level with approximately 1.4 microgram/cm^2 gap. This data showed the fluoride ion residue of 0.05% NaOH scrubbing for 12 min was significantly lower than current 0.02% NaOH scrubbing for 4 min.

From the above experiment results, it could be clearly shown that the removal of post-etched redeposit depended upon the concentration of NaOH and the scrubbing time. At higher concentration of NaOH and longer scrubbing time showed the part were cleaner by SEM and AFM images; and also the level of fluoride ions after clean has decreased. That can be explained by the increasing of the hydroxide ion presence to react with the residue.

The possible mechanism of this post-etched residue removal can be explained as follows. The primary role of NaOH in cleaning was the generation of hydroxide ions (OH^-). At higher concentration of NaOH, higher amount of OH^- was generated. This hydroxide ion (OH^-) would react with the post-etched redeposit (speculated to be AlF_3) to form aluminum hydroxide $Al(OH)_3$). To check the possibility of this mechanism, the cleaning solution after it was used to clean the part was colleted to analyze for the quantity of generated $Al(OH)_3$ by titrating with potassium fluoride (KF). The liberated lye was titrated and the solution was mixed with potassium fluoride. After the designated reaction time has passed, the titrated the lye was shown to liberate proportionally to aluminium content back with HCl. [9]

Based on the titration results, solution remained after cleaning with 0.05% NaOH contained higher $Al(OH)_3$ than solution of 0.02%NaOH. The $Al(OH)_3$ in the solution of 0.02%NaOH with 4 min scrubbing time; of 0.02%NaOH with 6 min of scrubbing time; and of 0.05%NaOH with 6 min of scrubbing time were 1.135 ppm, 1.343 ppm and 2.880 ppm, respectively. This can be explained that an increase in NaOH concentration and scrubbing time will increase the redeposit removal.

From abovementioned results, an increase in scrubbing time with a constant NaOH concentration would provide a higher $Al(OH)_3$. Similarly, an increase in NaOH concentration also resulted in the increasing amout of $Al(OH)_3$.

In term of improvement of spike defect reduction, this could be explained by the basic fundamental of photoresist stripping. The EDS could not detect the composition of spike because the size was too small. But it can be speculated that this defect contained the photoresist material since this was used during the etching process and might not be completely removed by the photoresist stripping prior to NaOH scrubbing process. As the photoresist was an acidic compound and the stripping process neutralized this acidity, in the process of neutralization the alkalinity of the resist stripper was consumed. Sodium is merely there along to neutralize the charge on the OH^-, acting as the active part of molecule to dissolve photoresist residue. The AFM images can show that cleaner parts are observed after clean with higher NaOH concentration and longer scrubbing time.

5.3. Electrical performance and failure comparison

In terms of confirming the proposed cleaning condition will result in a better performance of cleaner product, we have tested the read-write performance of the head. Figure 4.7 described the read-write head performance testing procedure.

5.3.1 Quasi-Static Test (QST) Yield

Figure 5.12 showed the result of processing with 0.05% NaOH scrubbing and scrubbing time of 12 min gave a higher yield than the current 0.02% NaOH with scrubbing time of 4 min scrubbing per the QST.

Process Flow	Sample size (Head)	Qty. Head pass specification	Quasi-static test yield
0.05%NaOH scrubbing 12 min	14478	12102	83.59%
Normal (0.02%NaOH scrubbing 4 min)	13852	11502	83.04%

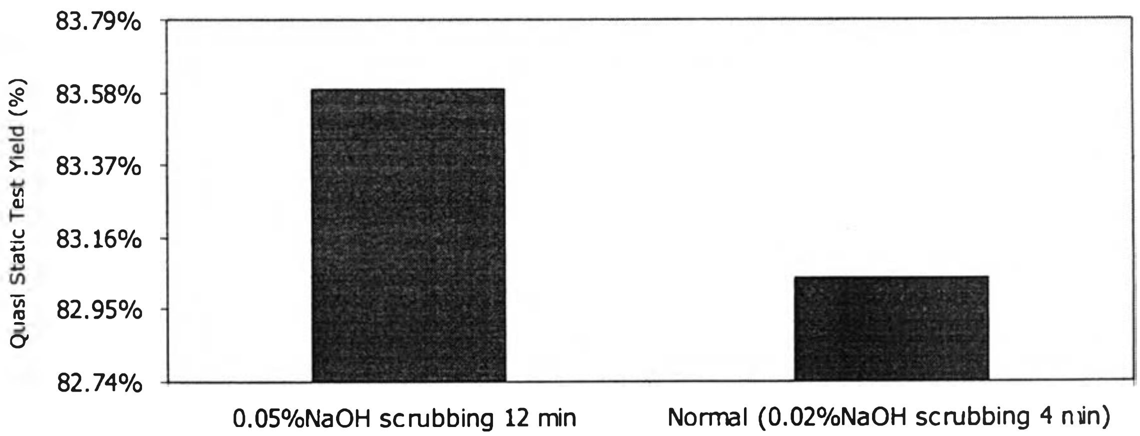


Figure 5.12. A comparison of the QST yield.

Test and CI for Two Proportions - Quasi-static test yield

```
Sample                                  X      N  Sample p
0.05%NaOH scrubbing 12 min          12102  14478  0.835889
Normal(0.02%NaOH scrubbing 4 min)   11502  13852  0.830349


Difference = p (1) - p (2)
Estimate for difference:  0.00553953
95% CI for difference:  (-0.00314746, 0.0142265)
Test for difference = 0 (vs not = 0):  Z = 1.25  P-Value = 0.211
```

Figure 5.13. A print-out of statistical analysis on QST yield from Minitab.

From the statistical analysis in Figure 5.13, the P-value of this analysis showed greater than 0.05 at 95%confidence level suggesting that in terms of potential percent defect of both groups are not different to each other considering in high confidence level with approximately 0.5% gap. In addition, this data showed that the electrical performance as the QST yield of 0.05% NaOH scrubbing 12 min was better than current 0.02%NaOH scrubbing 4 min.

5.3.2 Dynamic Electrical Test (DET) Yield

Figure 5.14 showed that the result of the new 0.05% NaOH scrubbing with 12 min scrubbing gave higher yield than current 0.02% NaOH scrubbing with 4 min scrubbing.

Process Flow	Sample size (Head)	Qty. Head pass specification	Dynamic electrical test yield
0.05%NaOH scrubbing 12 min	9264	7592	81.95%
Normal (0.02%NaOH scrubbing 4 min)	9048	7265	80.30%

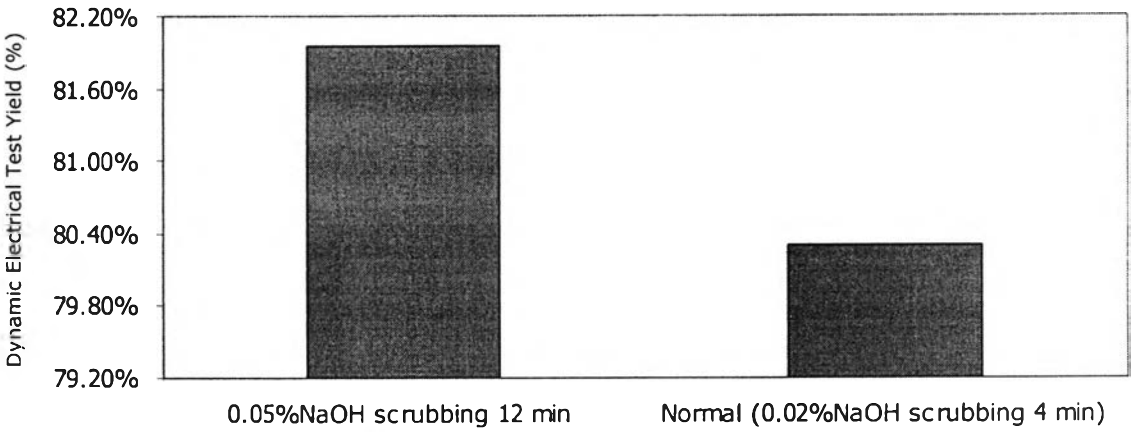


Figure 5.14. A comparison of DET yield.

Test and CI for Two Proportions - Dynamic electrical yield

```
Sample                                  X     N  Sample p
0.05%NaOH scrubbing 12 min           7592  9264  0.819516
Normal(0.02%NaOH scrubbing 4 min)    7265  9048  0.802940


Difference = p (1) - p (2)
Estimate for difference:  0.0165765
95% CI for difference:  (0.00524028, 0.0279128)
Test for difference = 0 (vs not = 0):  Z = 2.87  P-Value = 0.004
```

Figure 5.15. A print-out of statistical analysis on DET yield from Minitab.

From statistical analysis in Figure 5.15, the P-value of this analysis was less than 0.05 at 95%confidence level suggesting that in terms of potential percent defect of both groups are different to each other considering in high confidence level with approximately 0.16% gap. This data showed that the electrical performance as the DET yield of 0.05% NaOH scrubbing 12 min was better than current 0.02%NaOH scrubbing 4 min.

5.3.3 Early Touchdown (ETD) Failure During DET

ETD is a failure that occurred during DET with the applied voltage specification of 2.8 volts. This meant that any part that failed ETD criterion has failed because it was touching the magnetic disk with the applied voltage that was lower than 2.8 volt.The percent of ETD failure that was graded at 2.8 volts from new 0.05% NaOH scrubbing with 12 minutes scrubbing was lesser than current 0.02% NaOH scrubbing with 4 minutes scrubbing by almost 4 times. The %ETD of 0.05% NaOH process and 0.02% NaOH process was 0.99% and 3.86% respectively as shown in Figure 5.16.

Process Flow	Sample size	Early Touch Down (ETD) Fail	%ETD
0.05%NaOH scrubbing 12 min	9002	89	0.99%
Normal (0.02%NaOH scrubbing 4 min)	8828	341	3.86%

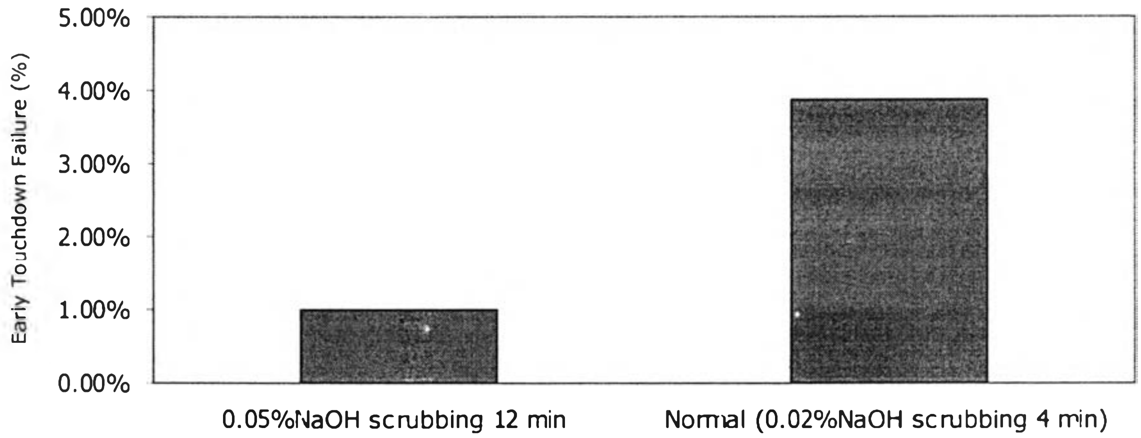


Figure 5.16. A comparison of ETD failure rate.

Test and CI for Two Proportions - ETD failure

```
Sample                                      X     N  Sample p
0.05%NaOH scrubbing 12 min                 89  9002  0.009887
Normal(0.02%NaOH scrubbing 4 min)         341  8828  0.038627

Difference = p (1) - p (2)
Estimate for difference:  -0.0287404
95% CI for difference:  (-0.0332500, -0.0242308)
Test for difference = 0 (vs not = 0):  Z = -12.49  P-Value = 0.000
```

Figure 5.17. A print-out of statistical analysis on ETD failure rate from Minitab.

From statistical analysis in Figure 5.17, the P-value of this analysis was less than 0.05 at 95%confidence level suggesting that in terms of potential percent defect of both groups are significantly isolated to each other considering in high confidence level with approximately 2.8% ETD reject. This data showed that the electrical performance in terms of %ETD failure of 0.05% NaOH scrubbing 12 min wa;; significantly lower than current 0.02% NaOH scrubbing 4 min.

5.3.4 Touchdown (TD) Limit Failure at Hard Disk Drive Assembly Operation

From the data shown in Figure 5.18. the failure rate of the new 0.05% NaOH scrubbing with 12 min scrubbing was lesser than current 0.02%NaOH scrubbing with 4 min scrubbing by about 0.5%- 1%.

Process Flow	Qty of Test (Hard Disk Drive)	Qty of Pass Test (Hard Disk Drive)	TD Limit Failure
0.05%NaOH scrubbing 12 min	2754	21	0.77%
Normal (0.02%NaOH scrubbing 4 min)	44913	656	1.46%

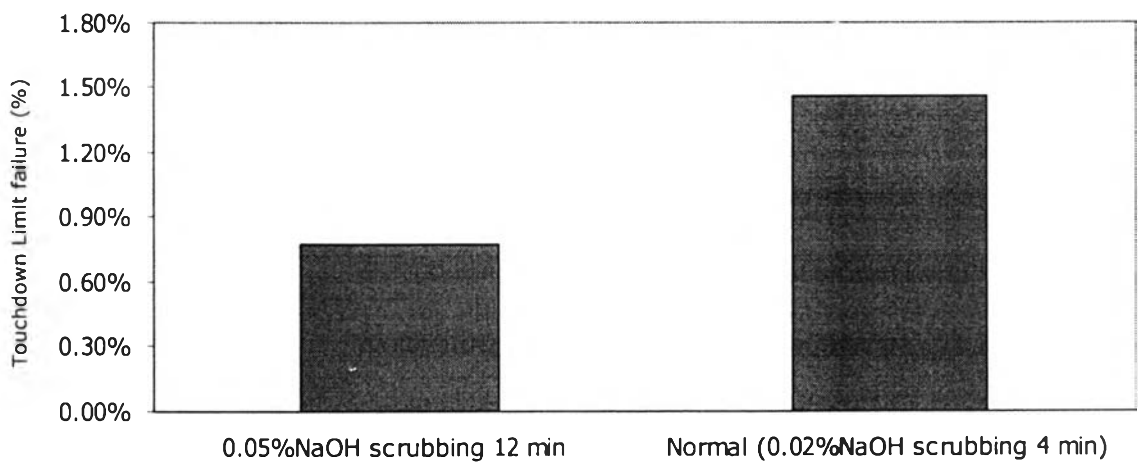


Figure 5.18. A comparison of TD failure.

Test and CI for Two Proportions - TD failure at Hard disk drive assembly

```
Sample                                  X      N  Sample p
0.05%NaOH scrubbing 12 min             21   2754  0.007625
Normal(0.02%NaOH scrubbing 4 min)     656  44913  0.014606


Difference = p (1) - p (2)
Estimate for difference:  -0.00698074
95% CI for difference:  (-0.0104138, -0.00354765)
Test for difference = 0 (vs not = 0):  Z = -3.99  P-Value = 0.000
```

Figure 5.19. A print-out of statistical analysis on TD failure rate from Minitab.

From the statistical analysis in Figure 5.19, the P-value of this analysis was less than 0.05 at 95%confidence level suggesting that in terms of potential percent defect of both groups are significantly isolated to each other considering in high confidence level with approximately 0.7% gap. This data showed that the electrical performance as the TD yield of 0.05% NaOH scrubbing 12 min is significantly better than current 0.02% NaOH scrubbing 4 min.

From above experimental results, it could be clearly that the new cleaning condition, 0.05%NaOH with 12 min scrubbing time, has improved the part cleanliness and giving a lesser defect rate. The reduction in the ETD and TD failure rate that are electrical in nature can be found to be related to the cleanliness of the part by the following argument. As the contamination level decreases, there was a lessening chance for these contaminants to loosen and dropped onto the magnetic media during read-write operation. This reduced the chances for disk scratches which will be occurred had it been otherwise. Therefore, the HDD in general will perform better by reducing the potential or failure probability of hard disk drive and it would also extend the lifetime of using hard disk drive which contains our valuable data.

5.4 Cost Analysis

The cost of making read-write head was considered and tabulated as shown in Table 5.4. The cost was calculated base on the increasing of sodium hydroxide usage and scrap cost due to ETD defect. Demand of read-write head per day is 1 million units. Therefore, the increasing of operating time was not affected.

Table 5.4. A comparison of operating cost and scrap cost.

	0.02%NaOH scrub 4 min	0.05%NaOH scrub 12 min
Unit per run	16,650	16,650
Cycle time per run (min) include rinsing&dry	8	16
Run per hour	7.5	3.75
Actual unit per day (total machine capacity)	2,997,000	1,498,500
Required unit per day **	**1,000,000**	**1,000,000**
NaOH consumption per run (lit)	0.5	1.0
NaOH consumption per run (g)	0.1	0.5
NaOH Usage (gram/ M unit)	**6.01**	**30.03**
NaOH cost per 100 g (baht)	500	500
NaOH cost per run (baht)	0.5	2.5
NaOH cost per M unit (Baht)	30.03	150.15
NaOH cost per M unit (USD)	**0.97**	**4.84**
ETD defect	3.86%	0.99%
ETD defect @ 1 K unit (scraped unit)	38,600	9,900
HGA cost (USD)	1.359	1.359
Scrap cost (base on 1M unit build) (USD)	**52,457.40**	**13,454.10**
Net cost for make 1 M unit {scrap cost USD + NaOH cost USD}	**52,458.37**	**13,458.94**

** demand of manufacturing per day = 1,000,000 unit

From abovementioned, the cost of current 0.02%NaOH with 4 minutes scrubbing are higher than 0.05%NaOH with 12 minutes scrubbing because of higher defect rate. At 1 million units built, 0.05%NaOH with 12 minutes can reduce cost approximately 39,000 USD.